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A GEL FOR LIQUID SCINTILLATION COUNTING BASED ON BITOLYLENE DIISOCYANATE

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A Gel for Liquid Scintillation Counting based on Bitolylene Diisocyanate

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LIQUID scintillation counting is useful in the radioassay of alpha and beta emitters in material insoluble in the scintillation solution. In order to avoid some of the difficulties associated with using finely divided silica as a gelling agent, BOLLINGER *et al.*[1] proposed the use of a diisocyanate-amine reaction product as a gelling mixture for a toluene-based scintillation solution. Although this mixture produced satisfactory gels, the use of toluene diisocyanate presents several serious problems; (1) The reactivity of the compound with atmospheric moisture results in short shelf-life under laboratory working conditions and (2) small amounts of volatile liquid, involving fractions of a milliliter, have to be pipetted routinely. The most serious problem is the toxicity of toluene diisocyanate and the attendant care required in handling it.

Experiments in this laboratory have shown that solid bitolylene diisocyanate, a moderately slow-reacting compound, was superior to toluene diisocyanate for gelling scintillation solutions. It was found in the course of routinely making and handling gelling mixtures for a large number of samples, the development of a rapid, relatively nontoxic system was desirable.

Experimental

The scintillation solution used in this study consisted of 7.0 g of 2,5-diphenyloxazole (PPO) and 1.5 g of p-bis-(o-methylstyryl)-benzene (bis-MSB) per liter of p-xylene. Armeen L-11 (Armour Industrial Chemical Company) was used as the amine component in producing the gels.

The bitolylene diisocyanate used in this study was Isonate 136T obtained from Polymer Chemicals Division of the Upjohn Company. Initial experiments were conducted to determine the effect of bitolylene diisocyanate concentration on the gel structure and on the quenching properties of the gel. Up to 500 mg of bitolylene diisocyanate were dissolved in 17 ml of the scintillation mix and subsequently reacted with 2 ml of the Armeen L-11 component.

Based on these experiments subsequent mixes were prepared by dissolving 10 g of bitolylene diisocyanate in a liter of the p-xylene scintillation solution and using 17 ml of this mixture for preparing individual samples. Addition of Armeen L-11 was reduced to 1 ml. Using this mixture, two beta emitters, ^{14}C and ^{147}Pm as insoluble compounds, were suspended using increasing weights of the compounds. All samples were counted on a Beckman room-temperature LS-200 liquid scintillation counter. Channel windows previously set for counting ^{3}H, ^{14}C, and ^{147}Pm in soluble samples were not changed for these experiments.

It was also found that a specific concentration range of reactants used in the gelling mixtures produced reversible gels which could be liquified by warming and gelled again by cooling. Gelled dispersions that were inhomogeneous were heated and redispersed when the mixture was liquified.

Table 1. *Efficiencies for tritiated gels containing Hexadecane-T*

Isonate 136T mg added	Counts/min*	Counting efficiency	Condition of gel†
0	7.27×10^4	0.32	Liquid‡
25	7.04×10^4	0.31	Thick; no gel
50	7.25×10^4	0.32	Reversible gel
75	7.23×10^4	0.32	Reversible gel
100	7.52×10^4	0.33	Reversible gel
250	7.76×10^4	0.34	Reversible gel
500	7.48×10^4	0.33	Not reversible

* 2.28×10^5 dis/min HDT added to each sample.
† Reversible to liquid state by warming.
‡ No reactants were added to first control sample.

Samples of bitolylene diisocyanate exposed to the atmosphere reacted with moisture, producing ureas which were insoluble in the scintillation mix. These insoluble compounds could be removed by stirring the solution after addition of the diisocyanate and filtering.

A sample of bitolylene diisocyanate on the shelf for approximately two years contained 10% insoluble material.

Results and discussion

Initially the quenching effect of the reacted gel was studied. A xylene soluble tritium compound was radio-assayed in order to show the extent, if any, of any quenching properties of the system. The effect of bitolylene diisocyanate on the counting efficiency for tritium in homogeneous gels is shown in Table 1.

Lithium carbonate labelled with ^{14}C was prepared for use in association with a project for the determination of environmental levels of ^{14}C.[2] Some of the labelled material was suspended in the gel and counted. The results are shown in Table 2.

Although a gradual decrease in counting efficiency was observed as the weight of $Li_2{}^{14}CO_3$ was increased, the loss in counting efficiency was more than offset by the increase in amount of ^{14}C. The net result is that the system of counting ^{14}C by this method is most efficient when counting 10 grams of $Li_2{}^{14}CO_3$.

Table 2. Gelled suspensions of $Li_2{}^{14}CO_3$

$Li_2{}^{14}CO_3$ Weight (g)	Counts/min*	Counting efficiency
1.0	2.24×10^4	0.91
2.0	4.09×10^4	0.83
3.0	5.83×10^4	0.79
4.0	7.99×10^4	0.81
5.0	9.39×10^4	0.76
6.0	10.6×10^4	0.72
7.0	12.1×10^4	0.70
8.0	12.8×10^4	0.65
9.0	12.8×10^4	0.58
10.0	13.5×10^4	0.55

* ^{14}C dis/min/g $Li_2{}^{14}CO_3 = 2.46 \times 10^4$.

Table 3. Gel suspension counting of ^{147}Pm*-tagged Yttrium oxalate*

Y Oxalate added (mg)	Counts/min*	Counting efficiency
5.0	2.83×10^3	0.64
10.0	5.37×10^3	0.61
20.0	10.4×10^3	0.59
50.0	21.4×10^3	0.48

* 1.77×10^4 dis/min ^{147}Pm/20 mg Y oxalate.

Yttrium oxalate tagged with ^{147}Pm was then prepared and gelled according to the method previously described. The results in Table 3 show a decrease in counting efficiency with increasing weight of solid; however, as in the case of $Li_2{}^{14}CO_3$, a net gain is achieved with increased activity.

Summary

An improvement has been made in the methodology for making gels used in liquid scintillation counting. The gel is based on the reaction product between bitolylene diisocyanate (Isonate 136-T and Armeen L-11 (branched aliphatic primary amine). The diisocyanate used has moderate reactivity and low vapor pressure. Gelling consistency and time can be varied with the concentration of diisocyanate dissolved in the scintillation solution.

Mention of commercial products used in connection with work reported in this article does not constitute an endorsement by the Public Health Service.

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